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1-(2-Hydroxy-5-methoxyphenyl)-3-methylbut-2-en-1-one[1]

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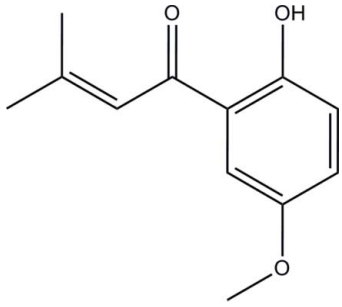
Key indicators: single-crystal X-ray study; T = 100 K, P = 0.0 kPa; mean σ(C–C) = 0.001 Å; R factor = 0.043; wR factor = 0.127; data-to-parameter ratio = 26.5.

The title compound, $C_{12}H_{14}O_3$, is a natural product derived from the medium-sized hawthorn *Crataegus persimilis* ('*prunipruni*folia'). The mean plane of the butene moiety is twisted by 13.27 (7)° with respect to the that of the dioxobenzaldehyde moiety. There is an intramolecular hydrogen bond between the hydroxyl group and the carbonyl O atom.

Related literature

For isolation from plant material, see: Castro *et al.* (1989). For the synthesis, see: Camps *et al.* (1985). For photolysis to form 4-chromanones, see: Primo *et al.* (1982). For a related structure, see: Zeller *et al.* (2010).



Experimental

Crystal data

$C_{12}H_{14}O_3$
M_r = 206.23
Monoclinic, $P2_1/c$
a = 14.027 (3) Å
b = 5.816 (1) Å
c = 12.829 (3) Å
β = 91.409 (8)°
V = 1046.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.45 × 0.37 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor 1997)
T_{min} = 0.959, T_{max} = 0.979
6425 measured reflections
3784 independent reflections
3179 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.127
S = 1.04
3784 reflections
143 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O3	0.87 (2)	1.74 (2)	2.523 (1)	149 (2)

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The purchase of the diffractometer was made possible by grant No. LEQSF (1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2405).

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[1] CAS Registry 84346–78–1.

supplementary materials

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1-(2-Hydroxy-5-methoxyphenyl)-3-methylbut-2-en-1-one

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Comment

The structure of title compound **I** can be described in terms of four planar moieties as defined by their constituent non-hydrogen atoms. The phenyl ring and three atoms bonded to it define the main molecular plane, with mean deviation of the defining atoms of $\delta_{r.m.s.}$ = 0.0145 (6) Å. With respect to this molecular plane, the mean plane of the carbonyl group (four atoms, $\delta_{r.m.s.}$ = 0.0044 (4) Å) and the plane of the methoxy group (three atoms) have dihedral angles of 2.50 (6)° and 4.33 (6)° respectively, while the mean plane of the butene moiety (four atoms, $\delta_{r.m.s.}$ = 0.0018 (4) Å) has dihedral angle 13.27 (7)°.

Experimental

Compound **I** was isolated as a natural product (Castro *et al.*, 1989). It has also been synthesized (Camps *et al.*, 1985). Suitable crystals were formed by very slow evaporation of a hexane solution over a period of three years.

Refinement

The positional and isotropic displacement parameters of hydroxyl atom H2A were refined independently. All other H atoms were placed in calculated positions, guided by difference maps, and refined as riding. Torsional parameters for the three methyl groups were refined, with C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C), while H atoms attached to sp^2 C atoms have C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C).

Computing details

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

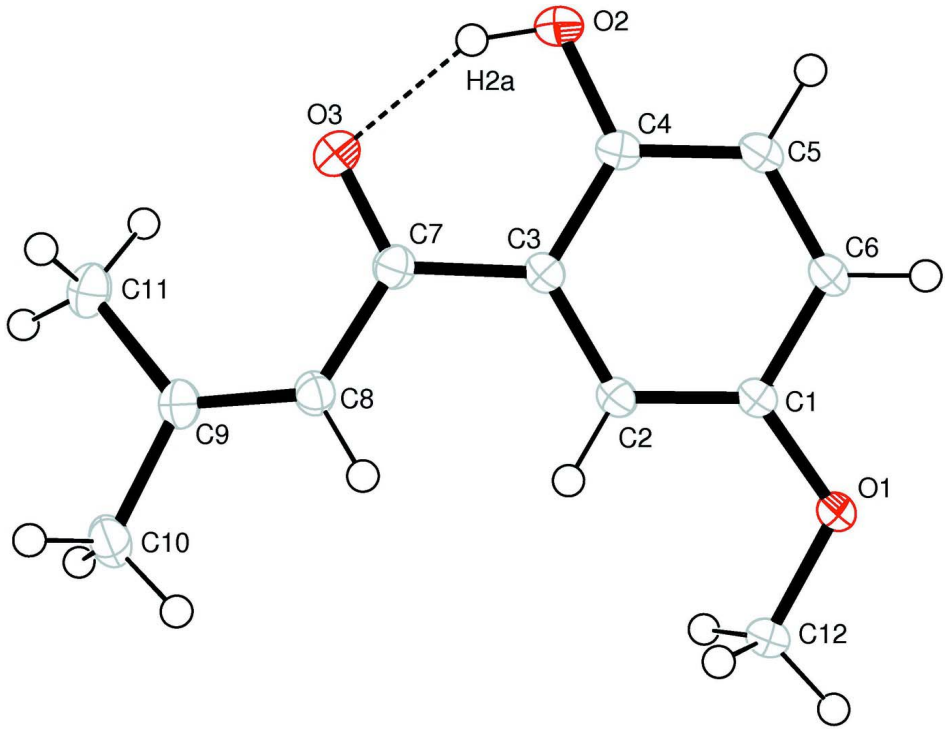

Figure 1

Molecular structure of (I) with displacement ellipsoids at the 50% probability level.

1-(2-Hydroxy-5-methoxyphenyl)-3-methylbut-2-en-1-one

Crystal data

$C_{12}H_{14}O_3$
$M_r = 206.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.027$ (3) Å
$b = 5.816$ (1) Å
$c = 12.829$ (3) Å
$\beta = 91.409$ (8)°
$V = 1046.3$ (4) Å^3
$Z = 4$
$F(000) = 440$
$D_x = 1.309$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3442 reflections
$\theta = 2.5$–32.6°
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Fragment, yellow
0.45 × 0.37 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: sealed tube
Horizonally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor 1997)
$T_{min} = 0.959$, $T_{max} = 0.979$
6425 measured reflections
3784 independent reflections
3179 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 32.6°$, $\theta_{min} = 3.2°$
$h = -21 \rightarrow 21$
$k = -8 \rightarrow 7$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.127$
$S = 1.04$
3784 reflections
143 parameters
0 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.2297P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.38220 (6)	1.03873 (14)	0.88580 (6)	0.01713 (16)
C2	0.31710 (6)	0.89827 (14)	0.93398 (6)	0.01652 (15)
H2	0.3003	0.9281	1.0040	0.020*
C3	0.27530 (6)	0.71019 (14)	0.87973 (6)	0.01641 (15)
C4	0.30238 (6)	0.66752 (15)	0.77617 (6)	0.01796 (16)
C5	0.36779 (6)	0.81313 (15)	0.72851 (6)	0.01992 (17)
H5	0.3853	0.7853	0.6586	0.024*
C6	0.40689 (6)	0.99610 (16)	0.78231 (6)	0.01999 (17)
H6	0.4509	1.0944	0.7491	0.024*
C7	0.20366 (6)	0.55840 (14)	0.92711 (7)	0.01901 (16)
C8	0.17084 (6)	0.60621 (15)	1.03279 (7)	0.01893 (16)
H8	0.1897	0.7485	1.0632	0.023*
C9	0.11620 (6)	0.46622 (15)	1.09083 (7)	0.01933 (16)
C10	0.08404 (7)	0.54666 (17)	1.19537 (7)	0.02467 (19)
H10A	0.1069	0.7037	1.2079	0.037*
H10B	0.1100	0.4446	1.2498	0.037*
H10C	0.0142	0.5442	1.1967	0.037*
C11	0.08255 (7)	0.23100 (16)	1.05970 (8)	0.02593 (19)
H11A	0.0197	0.2424	1.0251	0.039*
H11B	0.0781	0.1342	1.1219	0.039*
H11C	0.1279	0.1625	1.0117	0.039*
C12	0.40983 (6)	1.26466 (15)	1.03865 (7)	0.02072 (17)
H12A	0.3425	1.3041	1.0465	0.031*
H12B	0.4497	1.3925	1.0637	0.031*
H12C	0.4249	1.1263	1.0794	0.031*
O1	0.42786 (5)	1.22263 (11)	0.93148 (5)	0.02247 (15)
O2	0.26717 (5)	0.49087 (12)	0.71825 (5)	0.02388 (15)
H2A	0.2287 (12)	0.416 (3)	0.7580 (14)	0.052 (5)*
O3	0.17042 (5)	0.39337 (13)	0.87547 (6)	0.02755 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0188 (3)	0.0184 (3)	0.0143 (3)	−0.0008 (3)	0.0011 (3)	0.0000 (3)
C2	0.0168 (3)	0.0186 (3)	0.0142 (3)	0.0004 (3)	0.0009 (3)	0.0004 (3)
C3	0.0166 (3)	0.0175 (3)	0.0152 (3)	0.0007 (3)	0.0007 (3)	0.0006 (3)
C4	0.0195 (3)	0.0188 (3)	0.0156 (3)	0.0018 (3)	−0.0008 (3)	−0.0011 (3)
C5	0.0230 (4)	0.0234 (4)	0.0134 (3)	−0.0002 (3)	0.0018 (3)	−0.0006 (3)
C6	0.0226 (4)	0.0235 (4)	0.0140 (3)	−0.0026 (3)	0.0031 (3)	0.0010 (3)
C7	0.0181 (3)	0.0192 (4)	0.0198 (4)	−0.0002 (3)	0.0004 (3)	0.0002 (3)
C8	0.0179 (3)	0.0191 (3)	0.0199 (4)	−0.0006 (3)	0.0027 (3)	0.0002 (3)
C9	0.0157 (3)	0.0199 (4)	0.0224 (4)	0.0013 (3)	0.0007 (3)	0.0035 (3)
C10	0.0241 (4)	0.0267 (4)	0.0236 (4)	−0.0015 (3)	0.0071 (3)	0.0029 (3)
C11	0.0265 (4)	0.0208 (4)	0.0305 (5)	−0.0040 (3)	0.0023 (4)	0.0039 (3)
C12	0.0243 (4)	0.0223 (4)	0.0157 (3)	−0.0029 (3)	0.0026 (3)	−0.0027 (3)
O1	0.0286 (3)	0.0238 (3)	0.0153 (3)	−0.0094 (2)	0.0052 (2)	−0.0030 (2)
O2	0.0301 (3)	0.0225 (3)	0.0190 (3)	−0.0042 (3)	0.0017 (3)	−0.0051 (2)
O3	0.0317 (4)	0.0260 (3)	0.0251 (3)	−0.0101 (3)	0.0034 (3)	−0.0049 (3)

Geometric parameters (Å, º)

C1—O1	1.3708 (10)	C8—H8	0.95
C1—C2	1.3824 (11)	C9—C11	1.4982 (13)
C1—C6	1.4023 (12)	C9—C10	1.5003 (13)
C2—C3	1.4159 (11)	C10—H10A	0.98
C2—H2	0.95	C10—H10B	0.98
C3—C4	1.4127 (12)	C10—H10C	0.98
C3—C7	1.4795 (12)	C11—H11A	0.98
C4—O2	1.3540 (10)	C11—H11B	0.98
C4—C5	1.4002 (12)	C11—H11C	0.98
C5—C6	1.3751 (12)	C12—O1	1.4251 (11)
C5—H5	0.95	C12—H12A	0.98
C6—H6	0.95	C12—H12B	0.98
C7—O3	1.2499 (11)	C12—H12C	0.98
C7—C8	1.4690 (12)	O2—H2A	0.870 (18)
C8—C9	1.3538 (12)		
O1—C1—C2	125.22 (7)	C8—C9—C11	125.55 (8)
O1—C1—C6	114.77 (7)	C8—C9—C10	119.40 (8)
C2—C1—C6	120.00 (8)	C11—C9—C10	115.05 (8)
C1—C2—C3	120.46 (7)	C9—C10—H10A	109.5
C1—C2—H2	119.8	C9—C10—H10B	109.5
C3—C2—H2	119.8	H10A—C10—H10B	109.5
C4—C3—C2	118.71 (7)	C9—C10—H10C	109.5
C4—C3—C7	118.86 (7)	H10A—C10—H10C	109.5
C2—C3—C7	122.43 (7)	H10B—C10—H10C	109.5
O2—C4—C5	116.96 (8)	C9—C11—H11A	109.5
O2—C4—C3	123.13 (8)	C9—C11—H11B	109.5
C5—C4—C3	119.91 (8)	H11A—C11—H11B	109.5
C6—C5—C4	120.43 (8)	C9—C11—H11C	109.5

C6—C5—H5	119.8	H11A—C11—H11C	109.5
C4—C5—H5	119.8	H11B—C11—H11C	109.5
C5—C6—C1	120.48 (8)	O1—C12—H12A	109.5
C5—C6—H6	119.8	O1—C12—H12B	109.5
C1—C6—H6	119.8	H12A—C12—H12B	109.5
O3—C7—C8	120.88 (8)	O1—C12—H12C	109.5
O3—C7—C3	119.26 (8)	H12A—C12—H12C	109.5
C8—C7—C3	119.85 (7)	H12B—C12—H12C	109.5
C9—C8—C7	126.04 (8)	C1—O1—C12	117.01 (7)
C9—C8—H8	117	C4—O2—H2A	106.4 (12)
C7—C8—H8	117		

O1—C1—C2—C3	−178.95 (8)	C2—C1—C6—C5	−0.92 (13)
C6—C1—C2—C3	0.33 (13)	C4—C3—C7—O3	1.59 (12)
C1—C2—C3—C4	0.82 (12)	C2—C3—C7—O3	−179.06 (8)
C1—C2—C3—C7	−178.54 (8)	C4—C3—C7—C8	−176.96 (7)
C2—C3—C4—O2	179.35 (7)	C2—C3—C7—C8	2.39 (12)
C7—C3—C4—O2	−1.28 (12)	O3—C7—C8—C9	11.11 (14)
C2—C3—C4—C5	−1.39 (12)	C3—C7—C8—C9	−170.37 (8)
C7—C3—C4—C5	177.99 (8)	C7—C8—C9—C11	2.89 (14)
O2—C4—C5—C6	−179.87 (8)	C7—C8—C9—C10	−176.50 (8)
C3—C4—C5—C6	0.82 (13)	C2—C1—O1—C12	3.14 (12)
C4—C5—C6—C1	0.34 (13)	C6—C1—O1—C12	−176.18 (8)
O1—C1—C6—C5	178.43 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3	0.87 (2)	1.74 (2)	2.523 (1)	149 (2)